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Your reference
GS/AMW/P50062

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Request for grant of a Patent

Form 1/77

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2 Do not give trading styles, for example, 'Trading as XYZ company', nationality or former names, for example, 'formerly (known as) ABC Ltd' as these are not required.

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1 Title of invention

1 Please give the title of the invention

ELECTRODE SYSTEM

2 Applicant's details

❑ **First or only applicant**

2a If you are applying as a corporate body please give:

Corporate name
IMCO (1097) LIMITED

Country *(and State of incorporation, if appropriate)*

2b If you are applying as an individual or one of a partnership please give in full:

Surname

Forenames

2c In all cases, please give the following details:

Address
HALE TOP HOUSE EAST
THORLEY LANE
MANCHESTER AIRPORT
MANCHESTER

UK postcode *(if applicable)* M90 5UG

Country UNITED KINGDOM

ADP number *(if known)* 7423463001

2d, 2e and 2f:
If there are further applicants please provide details on a separate sheet of paper.

☐ **Second applicant (if any)**

2d If you are applying as a corporate body please give:

Corporate name

Country *(and State of incorporation, if appropriate)*

2e If you are applying as an individual or one of a partnership please give in full:

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Forenames

2f **In all cases**, please give the following details:

Address

UK postcode
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Country

ADP number
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3
An address for service in the United Kingdom must be supplied.
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3 Address for service details

3a Have you appointed an agent to deal with your application?

Yes [X] No [ ] ➡ ***go to 3b***

⬇
Please give details below

Agent's name
URQUHART-DYKES & LORD

Agent's address
TOWER HOUSE
MERRION WAY
LEEDS

Postcode LS2 8PA

Agent's ADP number 1644004

3b:
If you have appointed an agent, all correspondence concerning your application will be sent to the agent's United Kingdom address.

3b If you have not appointed an agent please give a name and address in the United Kingdom to which all correspondence will be sent:

Name

Address

Postcode

ADP number
(if known)

Daytime telephone numb r *(if available)* 1132452388

4 Reference number

4 Agent's or applicant's reference number *(if applicable)* GS/AMW/P50062

5 Claiming an earlier application date

5 Are you claiming that this application be treated as having been filed on the date of filing of an earlier application?

Please mark correct box

Yes ☐ No ☒ ➡ ***go to 6***

please give details below

☐ number of earlier application or patent number

☐ filing date *(day month year)*

☐ and the Section of the Patents Act 1977 under which you are claiming:

Please mark correct box

15(4) (Divisional) ☐ 8(3) ☐ 12(6) ☐ 37(4) ☐

6 Declaration of priority

6
If you are declaring priority from a PCT Application please enter 'PCT' as the country and enter the country code (for example, GB) as part of the application number.

Please give the date in all number format, for example, 31/05/90 for 31 May 1990.

6 If you are declaring priority from previous application(s), please give:

Country of filing	Priority application number *(if known)*	Filing date *(day,month,year)*

7

The answer must be 'No' if:
- *any applicant is not an inventor*
- *there is an inventor who is not an applicant, or*
- *any applicant is a corporate body.*

7 Inventorship

7 Are you (the applicant or applicants) the sole inventor or the joint inventors?

Please mark the correct box

Yes ☐ No ☒

A statement of Inventorship on Patents Form 7/77 will need to be filed (see Rule 15).

8

Please supply duplicates of claim(s), abstract, description and drawing(s).

8 Checklist

8a Please fill in the number of sheets for each of the following types of document contained in this application.

Continuation sheets for this Patents Form 1/77			
Claim(s)		Description	10
Abstract		Drawing(s)	3

Please mark correct box(es)

8b Which of the following documents also accompanies the application?

Priority documents *(please state how many)*	
Translation(s) of Priority documents *(please state how many)*	
Patents Form 7/77 - Statement of Inventorship and Right to Grant *(please state how many)*	
Patents Form 9/77 - Preliminary Examination/Search	
Patents Form 10/77 - Request for Substantive Examination	

9

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9 Request

I/We request the grant of a patent on the basis of this application.

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Date 18/05/1998
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ELECTRODE SYSTEM

This invention relates to an electrode system, and particularly, but not exclusively, to an electrode system suitable for use in preparative and analytical chemistry techniques.

Micro-electrode systems are extensively used in research. Such electrode systems are known as micro-electrode systems because the dimensions of the systems are on the micrometer scale. Such electrode systems provide very high field gradients and diffusion characteristics due to their small size. In addition, these types of electrode systems have found limited utility in bio-medical applications commercially and are typically used in, for example, blood gas analysis.

Electrode systems for preparative electrochemistry and electro analytical techniques depend crucially upon their geometry and the reproducibility of their manufacture for reliable operation.

Furthermore, the performance of such a system generally improves as the dimensions of the system are reduced which is why micro-electrode and even nanometre scale electrode systems are often used in these fields.

However, a disadvantage of known electrode systems of this type, is that the reproducibility and reliability of the fabrication process and the geometries which may be adopted become limited as the scale diminishes.

According to the present invention there is provided an electrode system comprising a laminated structure having at least one conducting layer;

at least one dielectric layer;

an aperture formed in the laminated structure; and

contact means for allowing electrical contact with at least one conducting layer.

The aperture may be in the form of a through hole which extends through the laminated structure and is open at either end.

Alternatively, the aperture may be in the form of a well having only one open end, the opposite end being closed and forming a well bottom.

At least one conducting layer of the system acts as an electrode on the internal wall of the hole or well. The electrodes may be treated as necessary to provide appropriate functionality, for example, pH measurement or surface treatment for electro-catalysis, for example.

Electrical fields are generated between the layers forming the laminated structure and within the hole or well to provide appropriate conditions within the laminated structure.

The electrode system according to the present invention may comprise a plurality of apertures formed within the laminated structure and spaced apart from one another.

An electrode system according to the present invention has many applications but could, for example, be used in the deionisation of a solution positioned on one side of a membrane forming the closed end of the well when the aperture is in the form of a well. In such a case, ions may be pumped through the electrode system as a consequence of a potential difference applied to the two electrodes on either side of the well bottom. In such a case, the well bottom would be formed from some form of ion exchange material, the ion flux being maintained by the applied potential difference.

Where the aperture is in the form of a through hole, the electrode system according to the present invention could be used in a preparative electrochemical system. In such a case, the reactants on one side of the electrode structure are passed through the structure or membrane using a pressure gradient, for example. As they pass through the holes, the reactants are modified by the applied electric field within each hole, thus

either producing product directly or generating intermediates which undergo further reaction to form the product.

The way in which the electrodes are treated to provide the appropriate functionality depends on the system in which the electrode system is to be used. If, for example, an electrode is required with a biological functionality for use in, for example, an enzyme or antibody system, a metal electrode would be treated with an organic conducting layer to prevent the activity of the biological agent from being destroyed.

Similarly, if a reference electrode is required, a silver electrode may preferably be used which may preferably be chloridised to form a silver / silver chloride reference electrode.

The laminated structure may comprise any number of conducting and dielectric layers, although preferably the laminated structure comprises three conducting layers.

The laminated structure may be built on silicon. This has the advantage of being optically flat. Alternatively, however, the laminated structure could be built on a material made from polymers.

The layers forming the laminated structure may be laid down using any one of a number of techniques including casting, spinning, sputtering or vapour deposition methods. The aperture may be mechanically or chemically introduced into the laminated structure. Advantageously, a micron gauge wire made of, for example, silver may be introduced into the laminated structure which wire may be etched out once the laminar structure has been completed. Alternatively, lithographic techniques may be employed. Additionally, physical techniques such as laser oblation and neutron annihilation may be used. It is possible to produce highly uniform electrode layers with precise separations using such techniques allowing highly reproducible functional structures to be obtained.

Advantageously, the dielectric layers are made from a rubbery material. This serves to amplify the effects of interspaced electrode systems as the resistance of the system is also effected by changes in the inter-electrode distance. During use of the electrode system, the rubber dielectric layers which separate electrode pairs swell thus changing the inter-electrode distance.

The electrode system according to the present invention may be in the form of a tape which is a substantially one-dimensional array or may be a multi-dimensional array, for example, in the form of a sheet or more complex matrix to enable repeated measurements with single use systems.

Preferably, the electrode system of the invention further comprises a micro heater structure incorporated into the system to control local conditions. Preferably, the micro heater is in the form of a resistive element laid down using semi-conductor techniques. The resistive element would provide localised heat.

Advantageously, the electrode system of the invention further comprises a piezo-electric vibrator.

The invention will now be further described by way of example only with reference to the accompanying drawings in which:

Figure 1a is a schematic representation of an electrode system according to the present invention incorporating a well formed in the laminated structure;

Figure 2 is a schematic representation of an electrode system according to the present invention incorporating a through hole formed in the laminated structure;

Figure 2a is a schematic representation of an electrode system according to the present invention having three electrodes and incorporating a well formed in the laminated structure;

Figure 2b is a schematic representation of an electrode system according to the present invention incorporating three

electrodes and incorporating a through hole formed in the laminated structure;

Figure 3 is a schematic three-dimensional representation of an electrode system according to the present invention incorporating two electrodes and a through hole formed in the laminated structure;

Figure 4a is a schematic representation of an electrode system according to the present invention incorporating two electrodes, a reagent loaded or functional dielectric, and a well in the laminated structure;

Figure 4b is a schematic representation of an electrode system according to the present invention incorporating two electrodes, a reagent loaded or functional dielectric, and a through hole formed in the laminated structure;

Figures 5a and 5b are schematic representations of an electrode system according to the present invention incorporating a specialised or functionalised layer structure; and

Figure 6 is a schematic representation of an electrode system according to the present invention forming a membrane transport system.

Referring to Figure 1a, an electrode system according to the present invention is designated generally by the reference numeral 1. The electrode system 1 comprises a laminated structure 2 formed from alternating layers of conductor 3 and dielectric (or insulator) 4. The laminated structure illustrated comprises two conductor layers 3 and two insulating layers 4. The conductor layers 3 form electrodes in the electrode system 1.

The laminated structure 2 is formed on a base 5 which may be formed from a substrate formed from silicon, for example, or may be formed from a polymeric material.

The laminated structure has formed within it a well 6 in the form of an aperture being open at one end 7 and closed at the opposite end 8.

The electrode system shown in Figure 1b is similar to that shown in 1a and corresponding reference numerals have been used in Figure 1 for ease of reference. The electrode system 10 shown in Figure 1b has formed within the laminated structure 2 a through hole 11 rather than a well 7. There is, therefore, no base 5, and instead the electrode system 10 comprises three layers of dielectric 4.

Referring now to Figures 2a and 2b electrode systems 20 and 30 respectively are illustrated which electrode systems 20, 30 are similar to the electrode systems 1, 10 respectively. Like parts have been designated with similar reference numerals to avoid confusion. Each of the electrode systems 20, 30 comprises three electrode layers rather than two as shown in the systems depicted in Figures 1a and 1b respectively.

In fact, any number of electrode layers could be present in an electrode system according to the present invention.

Hole 11 or well 7 formed in the electrode according to the present invention and formed through the laminated structure 2 is thus formed from alternating layers of insulating and conducting materials. This produces a circular micro-band electrode system within a tube. This can be seen more clearly with reference to Figure 3 which is a three-dimensional representation of the electrode system 10 as shown in Figure 1b.

The shape of the hole or well may be varied to provide non-uniform fields if required for the application to which the system is to be put.

The electrodes 3 within the hole or well structure may subsequently be processed in order to provide the required functionality whether that be chemical and / or electro-chemical and / or physical modifications.

If, for example, the electrode system was required to have biological functionality for use in an enzyme or antibody system, for example, the electrodes would be formed from metal and would first have to be treated with an organic conducting

layer to prevent the activity of the biological agent from being destroyed.

Similarly, if an electrode system was required to act as a reference electrode, the electrodes 3 would be formed from silver which would then be chloridised to form a silver / silver chloride reference electrode.

Typically, however, the electrodes would be formed from flashed gold. The gold can readily be sputtered onto a polymer which would act, both as the mechanical support and as the dielectric layer 4. Any form of polymer or other dielectric material which is capable of acting as a support may be used. Similarly, any suitable conductor, for example, a noble metal, PET (polyethylenetetraphthalate) would make a good dielectric. Other specialised materials such as ion exchange polymers, for example, cation doped polystyrene sulphonate could be used for specialised applications.

The dimensions of the layers 3, 4, and holes or well 7, 11 forming the electrodes system may be varied both in terms of the diameter, shape and the depth of the hole or well. Several micron thicknesses of layers, and of holes or well diameters are achievable. The dimensions of the electrode system depend upon the materials used and the techniques employed to form the electrode system.

Typically, the diameter of the hole or well will be one micron. Each layer 3, 4 will be approximately one micron.

As the dimensions in the electrode system according to the present invention are extremely small, the fields generated within the structure are exceptional, and enable highly efficient measurement and / or modification of materials entering into or passing through such structures. Such structures are simple to manufacture to extremely high tolerances. In addition the structures have extremely low dead volume thus considerable simplifying physical sampling regimes.

At a location remote from the hole 7 or well 11 a means to enable electrical contact with each of the conducting layers

3 is provided. One such means of providing electrical contact would be to slice back the outer edges of the dielectric layers 2 thus exposing the extreme ends of each of the dielectric layers 3. These exposed ends allow electrical contact to be made. Each hole or well may be individually addressable in which case each hole or well may have a different function. Alternatively, groups of holes or wells or all of the holes or wells in a structure may be addressed in parallel enabling amplification of signals and parallel material processing. This latter arrangement may be required for larger scale synthetic applications.

When an electrode system according to the present invention is used in a mass transport system the potential difference created causes diffusion of desired chemical species to the hole or well.

In some cases, for whatever reason, this process is slow and the mass transport may be aided, for example, through use of a piezo-electric vibrator or an ultra-sonic probe.

Mass transport may be additionally controlled where required by conventional microscopic means used in electrochemistry. These techniques include membrane and diffusion, wall jet / wall pipe techniques, rotation, vibration etc. In the case of an electrode system having a through hole, the mass flow may additionally be controlled using differential pressure techniques.

Materials passing into the structure may be pre-treated. A system suitable for pretreatment of material is shown in Figures 4a and 4b, where parts equivalent to those in Figures 1a and 1b respectively have been given equivalent reference numerals.

The electrode system 50, 60 shown in Figures 4a and 4b respectively contain two electrode layers 3, two dielectric layers 4 and a reagent loaded or functional dielectric layer 5. The reagent loaded or functional dielectric layer 12, is able to provide additional functionality by providing ions or other

materials, for example, ensure the reproducible behaviour of subsequent systems within the structure. Ions, for example, could be provided by ion exchange resin materials. Other matrices could be employed to provide co-factors for biosensors etc. The layer 12 could act as a buffer if, for example, there was some kind of ion exchange taking place where a remote reservoir was replenishing the ions exchanged within the medium in contact with the membrane.

Referring now to Figure 5a and 5b, an electrode system according to the present invention is designated generally by the reference numeral 70. Parts equivalent to those shown in Figure 1b have been given equivalent reference numerals.

The electrode system 70 comprises a specialised layer 13 between the two electrode layers 3. The system further comprises means 14 to produce physical or chemical gradients or potentials to the specialised layer 13. The specialised layer 13 may be in the form of, for example, an ion exchange resin, gel or solid electrolyte. In such a system 70 mass transport from one lateral region of the structure to another may be effected by, for example, osmosis, electro-osmosis, electrophoresis, electrochromatography, ion migration etc. Reverse flow and counter current techniques may be employed to effect changes in process flows, including deionisation, for example.

The dielectric layers 4 may be formed from a rubbery material. A suitable material would be a polymer which swells when molecules of, for example, water enter the solid state matrix of the polymer. In a system incorporating rubbery dielectric layers, when the layer swells, the spacing between the electrode layers 3 increases and thus the resistance measured across the electrodes would in most cases increase. Thus, the interspaced electrodes may simply be interrogated to determine the degree of swelling of the dielectric layers function of the observed resistance.

In more complicated systems, material may be grown between the electrode layers and the rubber dielectric layers, and the stress placed on the material as a consequence of the swelling of the dielectric layers may be measured.

Referring now to Figure 6, an electrode system according to the present invention suitable for use in deionisation of the solution designated generally by the reference numeral 80. The electrode system 80 comprises a plurality of holes 82. Each of the holes 82 is split in two by the presence of a special layer 84 which serves as the well bottom. The well bottom is formed from an ion exchange material. The ion flux is maintained by the applied potential difference.

Drawings

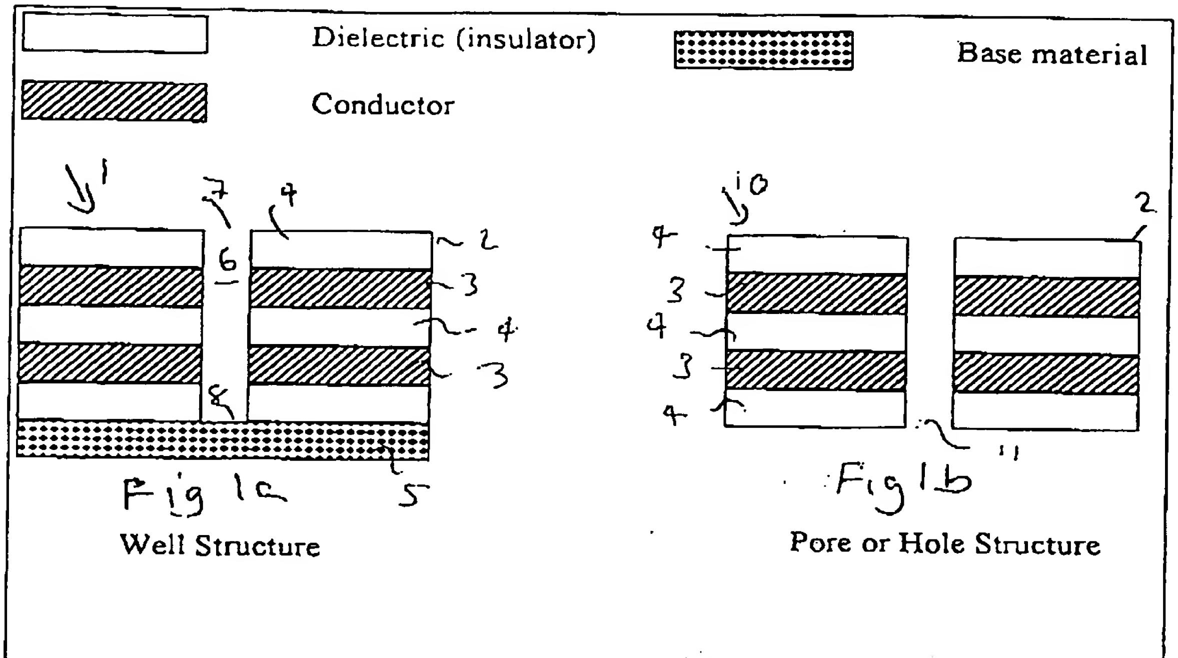

Figure 1. Two electrode systems

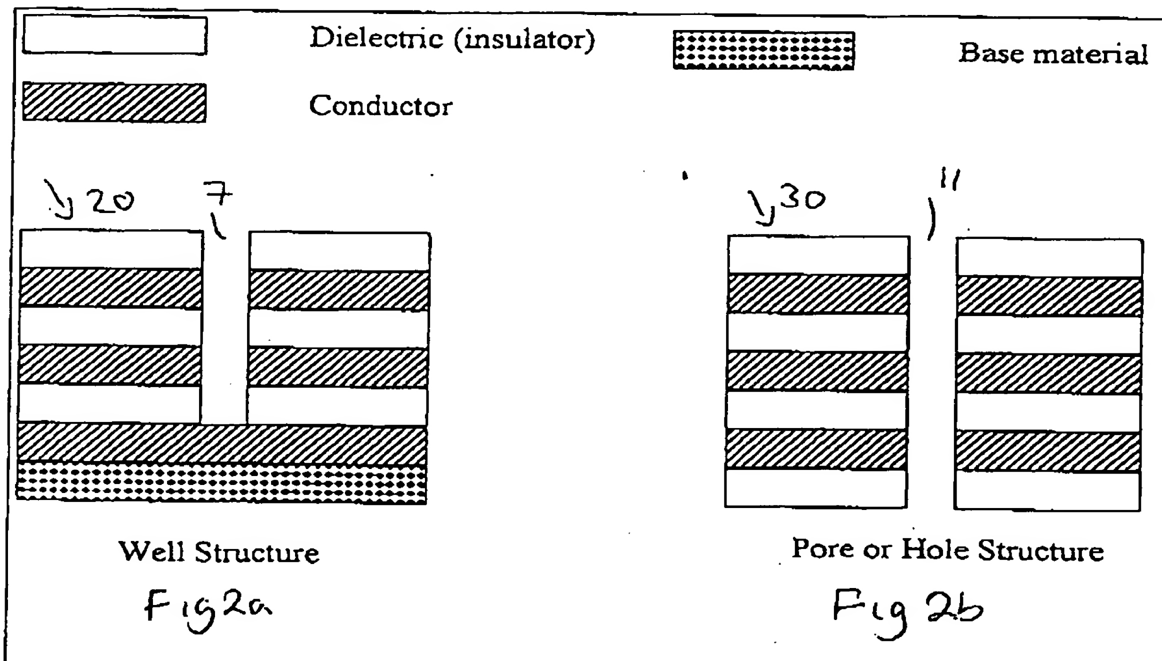

Figure 2. Three electrode systems

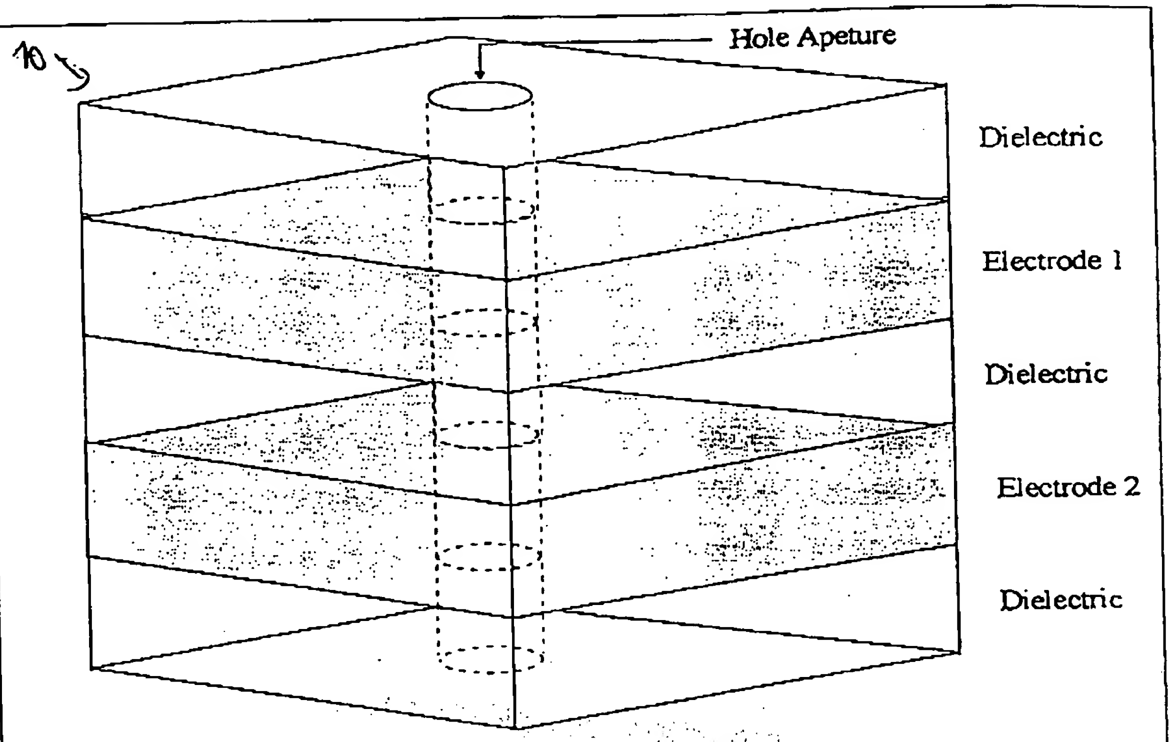

Figure 3. Three dimensional representation of two electrode hole structure

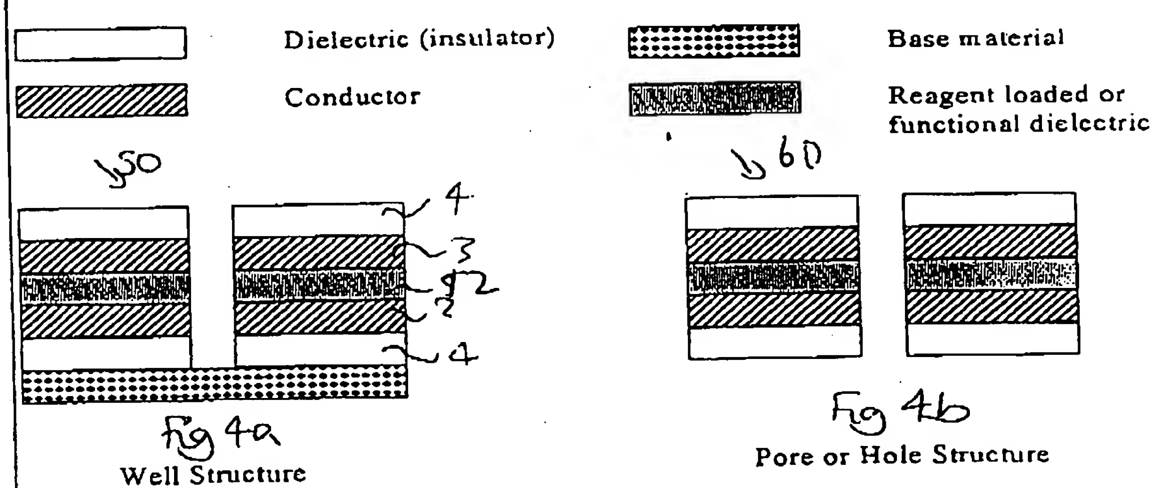

Figure 4. Reagent/functionalised two electrode systems

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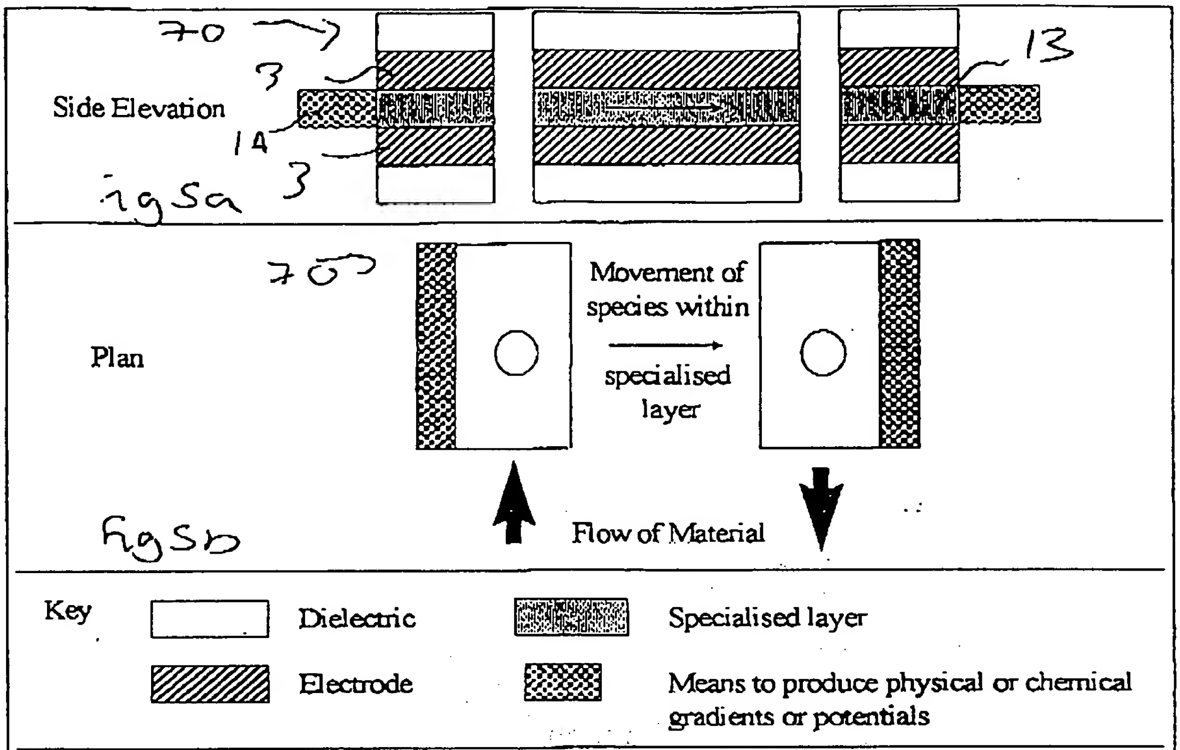
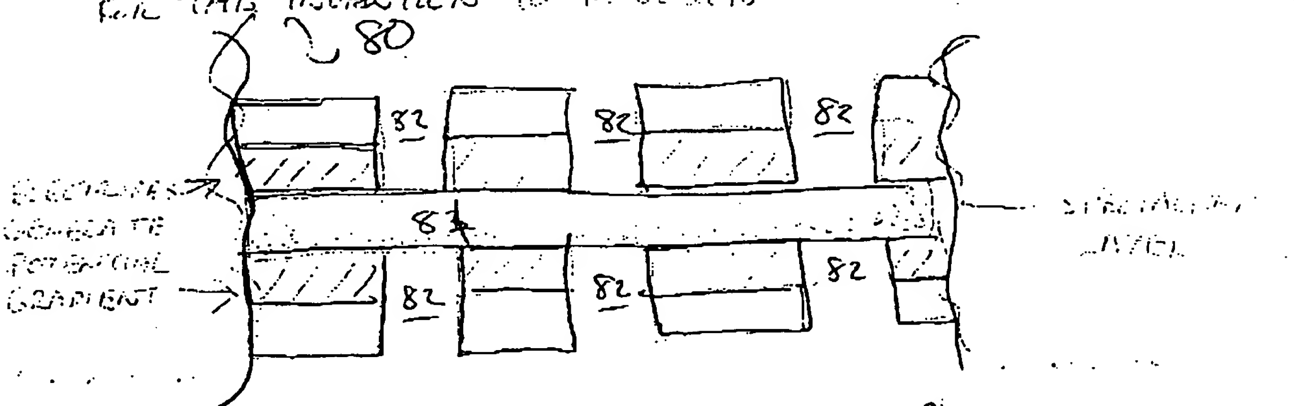


Figure 5. Specialised or Fucntionalised Layer Structures

NOTE: THE HOLES NEED NOT BE ADJACENT TO EACH OTHER FOR THIS INVENTION TO FUNCTION



POTENTIAL GRADIENT FORCES SPECIES (IONS FOR EXAMPLE) TO MOVE ACROSS THE MEMBRANE, THIS MIGHT BE USED FOR EXAMPLE TO DE-IONISE A WATER SUPPLY.

Figure 6 Membrane ~~[illegible]~~ Transport system

P[illegible] GB99 [illegible]
18.5.99
VDL